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Effects of Fiber Coatings on Tensile Properties of Hi-Nicalon SiC/RBSN Tow Composites

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ABSTRACT

Uncoated Hi-Nicalon silicon carbide (SiC) fiber tows and those coated with a single surface layer of pyrolytic boron nitride (PBN), double layers of PBN/Si-rich PBN, and boron nitride (BN)/SiC coatings deposited by chemical vapor deposition (CVD) method were infiltrated with silicon slurry and then exposed to N_2 for 4 hr at 1200 and 1400 °C. Room temperature ultimate tensile fracture loads and microstructural characterization of uncoated and CVD coated Hi-Nicalon SiC fiber reinforced reaction-bonded silicon nitride (RBSN) tow composites were measured to select suitable interface coating(s) stable under RBSN processing conditions. Results indicate that room temperature ultimate fracture loads of the uncoated Hi-Nicalon SiC/RBSN tow composites nitrided at both temperatures were significantly lower than those of the uncoated Hi-Nicalon tows without slurry infiltration. In contrast, all CVD coated Hi-Nicalon SiC/RBSN tow composites retained a greater fraction of the dry tow fracture load after nitridation at 1200 °C, but degraded significantly after nitridation at 1400 °C. Reaction between metal impurities (Fe and Ni) present in the attrition milled silicon powder and uncoated regions of SiC fibers appears to be the probable cause for fiber degradation.

[Key words: SiC fibers, interface coatings, nitridation, Fe-Ni silicide reaction, microstructure]

INTRODUCTION

The SiC fiber reinforced reaction-bonded silicon nitride matrix (SiC/RBSN) composite system is considered as a potential material for high temperature applications because of its key processing and property advantages. First, SiC fiber is chemically compatible with the silicon nitride (Si_3N_4) matrix. Second, RBSN processing is a low cost, near net shape process which can be adapted to the fabrication of complex shaped components. Third, the processing conditions for RBSN can be tailored to avoid strength and microstructural degradation in SiC fibers and the fiber/matrix interface, and to achieve desirable composite properties. Fourth, RBSN is a creep resistant material under non-oxidizing conditions and exhibits adequate strength for high temperature applications.

Previous studies have shown that strong and tough SiC/RBSN composites can be fabricated by reinforcing RBSN with 144 μm diameter chemically vapor deposited (CVD) SiC fibers [1,2]. Although used as a model system to correlate constituent microstructure with mechanical and thermal properties, this composite could not be used for high temperature applications for two reasons. First, the large diameter SiC fibers cannot be bent to a radius less than 12 mm. This severely restricts this composite to fabrication of simple shapes. Second, machining complex shaped components from a composite block is prohibitively expensive. In addition, machining invariably causes damage to the fibers and exposes the fiber ends to the environment which can lead to significant property degradation. Both of these problems can be overcome by choosing small diameter (~14 μm) SiC fibers as reinforcement and developing a near net shape RBSN processing method.

Several attempts have also been made previously to reinforce RBSN matrix with small diameter SiC fibers [2-4]. Although these studies demonstrated strain capability beyond matrix cracking strength, the ultimate strength of the composites was low primarily due to strength degradation of the coated SiC fibers during high temperature processing of the composites. In recent years significant improvements in high temperature strength and microstructural stability have been accomplished in small diameter SiC fibers derived from polymer precursor methods. These SiC fibers such as Hi-Nicalon and Hi-Nicalon-S from Nippon Carbon, and Sylramic from Dow Corning are commercially available. Because of their improved high temperature capability, these fibers allow deposition of interface coatings at higher temperatures, thus improving microstructural stability and properties of the coating, and use of high temperature processing approaches for composite fabrication.

In a preliminary study, Bhatt and Garg [5] have shown that uncoated Hi-Nicalon SiC fibers and those coated with a single layer of PBN and Si-rich PBN retained a large fraction (~90 percent) of their as-produced room temperature tensile strength after 400 hr exposure in N_2 at 1400 °C. However, the same coated fibers showed significant loss in strength when embedded in a silicon powder bed for 24 hr at 1400 °C and then tensile tested at room temperature. However a detailed microstructural and strength stability study of Hi-Nicalon SiC fibers with these and other commercially applied coatings under SiC/RBSN processing temperature range from 1150 to 1400 °C has not been fully investigated.

This study had three objectives: first, to determine strength and microstructural stability of Hi-Nicalon SiC fiber tows that were coated with either a single surface layer of pyrolytic boron nitride (PBN), double layers of PBN/Si-rich PBN, or boron nitride (BN)/SiC coatings by CVD, and that were infiltrated with silicon slurry and processed under RBSN conditions; second, to determine the mechanism of degradation of CVD coated Hi-Nicalon SiC fiber tows; third, to down select the most stable CVD coated Hi-Nicalon fibers for future RBSN composite processing.

EXPERIMENTAL VARIABLES

Uncoated Hi-Nicalon SiC fiber spools were procured from Dow Corning Company, Midland, Michigan. The fiber tow in the spool contained ~500 filaments. The diameter of individual fibers in the tow varied from 8 to 16 μm with an the average diameter of ~14 μm. Three types of CVD coating were examined; a single layer of pyrolytic boron nitride (PBN) ~1 μm thick, a dual layer of 0.8 μm PBN/0.2 μm Si-rich PBN, and a dual layer of 0.8 μm boron nitride (BN)/0.2 μm SiC. The PBN and PBN/Si-PBN coatings were applied by Advanced Ceramics Corporation, Cleveland, Ohio and the BN/SiC coating by 3M, St. Paul, Minnesota.

The silicon powder required for the silicon slurry was obtained from Union Carbide Company. The as-received powder contained particles ranging in diameter from 0.7 to 30 μm, with an average particle diameter of 4.9 μm. The powder also contained 0.07 wt% Al and 0.5 wt% Fe as major metallic impurities. The as-received powder particle size was too large to infiltrate within the fiber tow. Therefore, attrition milling was used to reduce the particle size. The as-received silicon powder was blended with 3 wt% NiO and then wet attrition milled in a stoddard solvent for 48 hr in a Si_3N_4 container using Si_3N_4 grinding media. The attrition milling procedure used was similar to that described in reference 6. After attrition milling, the bulk of the stoddard solvent was removed by filtering the silicon powder/stoddard mixture through a porous filter. The excess stoddard solvent still remaining in the silicon powder was

removed by drying for 24 hr in a vacuum oven at 500 °C. The dried powder was stored in a glove chamber filled with high purity N_2.

Impurity contents, particle size range, and specific surface area of silicon powders were determined by wet chemistry, laser light scattering (Microtrac, Model 7991), and the three point Brunauer-Emmett-Teller (BET) adsorption (Micromeritics, Model ASAP 2010) techniques, respectively.

The silicon slurry used for infiltrating into the tow specimens was prepared by ball milling a mixture of ~30 wt% attrition milled silicon powder, polybutylmethyl acrylate (PBMA), 4 wt% dibutyl pthalate, 1 wt% fish oil, and 60 to 65 wt% isopropyl alcohol for 3 to 4 hr in a polyethylene bottle filled with Si_3N_4 grinding balls similar to those used for the attrition milling. After ball milling, the required amount of the homogenized slurry was poured into a test tube. For slurry infiltration, both the CVD coated as well as uncoated Hi-Nicalon fiber tows were cut into 150 mm long pieces. The polyvinyl alcohol (PVA) sizing present on the surface of uncoated fibers was removed by using isopropyl alcohol. Each piece was first manually dipped into the slurry and then slowly pulled out of the test tube such that the slurry infiltrated and coated the fiber tow. After slurry coating, tows were dried in air. At least 15 tows of each uncoated and CVD coated Hi-Nicalon SiC fiber tow specimens were prepared.

For tensile tests, each slurry coated fiber tow was further cut into 50 mm lengths. Individual tow specimen was placed at the center of a rectangular picture frame cut from an index card. Approximately 12.5 mm of the fiber tow from each end was bonded to the short side of the picture frame using a 15 min curing epoxy leaving a 25 mm gauge length. After curing, the picture frame was aligned and gripped in a tensile testing machine and the long sides of the picture frame were cut. The fiber tow specimens were pulled at a constant rate of 1.3 mm/min. At least 10 tow specimens were tested for each condition.

Metallographic specimens of the tow composites were sectioned normal to the fiber, lapped on diamond impregnated metal discs and polished on a vibratory polisher. Polished specimens were sputter coated with a thin layer (~10 nm) of palladium and then examined in a JEOL 840A scanning electron microscope (SEM). For preparation of transmission electron microscope (TEM) specimens, a procedure similar to that described in reference 7 was used. To describe the procedure briefly, the nitrided tow composites were cast and vacuum degassed in a mixture of epoxy and 6 μm alumina powder in a 3 mm diameter by 20 mm long brass tube. After curing at 130 °C for 15 min, ~1 mm thick disks were sectioned using a diamond saw. The disks were mechanically ground from both sides to 120 μm, dimpled

50 μm from each side and ion-beam thinned to perforation using argon jet. A Phillips Model 400T transmission electron microscope (TEM) operating at 120KV was used for bright field imaging and convergent beam electron diffraction (CBED). The TEM was equipped with an energy dispersive x-ray spectrometer (XEDS) for chemical identification.

RESULTS AND DISCUSSION

After 48 hr attrition milling, the average particle size of the as-received silicon powder was reduced from 4.9 to 0.5 μm and the nominal surface area increased from 4.9 to 70 m^2/gm. Impurity analysis results indicate that the as-received silicon powder contained iron and oxygen as major impurities: ~ 0.5 wt% Fe and 0.4 wt% O_2. The 48 hr attrition milled silicon powders also contained 0.4 wt% yttrium, 0.1 wt% aluminum and 9 wt% oxygen impurities. The yttrium and aluminum impurities came from Si_3N_4 grinding media which contained 6 wt% Y_2O_3 and 2 wt% Al_2O_3 as sintering additives.

A SEM photograph of the transverse cross section of the PBN/Si-rich PBN-coated Hi-Nicalon/RBSN tow composite specimens indicating distribution of fibers within the tow is shown in figure 1. The cross section of the RBSN infiltrated tows is highly irregular and contained voids in the interior of the tow, but the silicon slurry and hence Si_3N_4 is well infiltrated between the fibers in the tow. The distribution of fibers in the tow composite specimen is reasonably uniform. All other coated tow composites showed similar features. A higher magnification photograph of the area shown in figure 1 reveals considerable variation in CVD coating thickness on individual fibers within the fiber tow (fig. 2); the outer fibers had greater thickness of CVD coating than the inner fibers. SEM photographs of the cross-section of PBN, PBN/Si-rich PBN, and BN/SiC coated Hi-Nicalon SiC fibers in RBSN matrix tow composites processed at 1400 °C are shown in figure 3. Although morphology of the coating varies in all three types of coating, interface coating is seen on most of the fibers. In the case of BN/SiC coated Hi-Nicalon SiC fiber tows, the SiC coating is nodular and irregular, but by far the BN layer of the BN/SiC coating is more uniform than the PBN layer on the other two coating combinations.

Because of the irregular cross-section of the tow composite specimens calculation of cross-sectional area became difficult. Therefore, in generating tensile data, the ultimate fracture load, instead of ultimate tensile strength (UTS), is reported. Furthermore it is also assumed that RBSN matrix does not contribute to ultimate fracture load of the tow composites. This is a valid assumption because the as-fabricated tow composite specimens showed cracks normal

to the testing direction. In general, in ceramic matrix composites it is known that the matrix fractures well before the fibers reach their ultimate fracture strength, and UTS of the composite is controlled to a large extent by the fiber bundles. Typical room temperature load-displacement curves of the uncoated and PBN/Si-rich PBN coated Hi-Nicalon SiC fiber dry tows, and the tows infiltrated with silicon and nitrided at 1200 °C and at 1400 °C for 4 hr in nitrogen are shown in figure 4. According to this figure, the room temperature fracture loads of the uncoated tow composites nitrided at 1200 °C and at 1400 °C are significantly lower than that of the dry bundles whereas the room temperature fracture loads of PBN/Si-rich PBN coated fiber tow composites decreased with increasing nitridation temperatures indicating progressive degradation of the fiber during the RBSN processing stage. The ultimate room temperature fracture loads of uncoated and all three types of coated tow composites before and after nitridation at 1200 °C and at 1400 °C for 4 hr are shown in figure 5. The error bars in the figure represent ± 1 standard deviation. In all cases, the ultimate fracture load decreased with increasing nitridation temperature. Both PBN/Si-rich PBN and BN/SiC coated Hi-Nicalon/RBSN tow composites nitrided at 1400 °C yielded nearly the same ultimate fracture loads within the experimental scatter.

TEM analysis of the tow composites was performed to determine phase stability of the coating and possible reaction between the SiC fiber and RBSN matrix, or between the interface coating and RBSN matrix. Results are shown in figures 6 to 10. Bright field TEM micrographs of the transverse cross-section of the uncoated Hi-Nicalon SiC fiber tow composite specimens nitrided at 1200 °C in N_2 for 4 hr show a solid state reaction between the Hi-Nicalon fiber and RBSN matrix (fig. 6(a)). The reaction zone thickness increases with increase in temperature of nitridation for a fixed nitridation time (fig. 6(b)). The average thickness of the reaction zone is ~0.1 and 0.5 μm after nitridation at 1200 and 1400 °C for 4 hr, respectively. XEDS analysis of the reaction zone indicates that it contains particles rich in iron and nickel. The presence of iron and nickel can be traced to the silicon powder and to nickel oxide(nitridation enhancing additive) used.

Coatings of PBN (fig.7) and PBN/Si-rich PBN (fig. 8) both provided protection of the Hi-Nicalon SiC fibers from the iron and nickel reaction. Both coatings consist of several layers. In some specimens these layers were debonded. It is not clear whether debonding occurred during specimen preparation or pre-existed. A small reaction thickness (0.2 μm at 1400 °C) was observed between each coating and the RBSN matrix. This reaction zone was also rich in iron and nickel. No reaction was observed between the Hi-Nicalon SiC fibers and PBN or PBN/Si-rich PBN

coatings. The nonuniform PBN and PBN/Si-rich PBN coatings of the fiber tow left regions of the fibers unprotected allowing the iron/nickel reaction with the fiber (fig. 8(b)).

A bright field image of the transverse cross-section of the BN/SiC coated Hi-Nicalon/RBSN tow composite nitrided in N_2 at 1200 °C is shown in figure 9(a). The crack in the BN/SiC coating, and the debonded region between the fiber and BN coating seen in figure 9(a) was probably caused during specimen preparation. Little reaction between the SiC coating and RBSN matrix was noticed. In contrast, the BN/SiC coated Hi-Nicalon/RBSN tow composites nitrided at 1400 °C showed almost complete reaction of the SiC coating with the RBSN matrix (fig. 9(b)). The reaction zone contained particles which are rich in iron and nickel impurities similar to that observed in specimens of the uncoated Hi-Nicalon/RBSN tow composites. Convergent beam electron diffraction (CBED) identified the reaction product as an iron silicide (Fe_2Si) with the CsCl structure and a lattice parameter of 0.294 nm (fig. 10). Since XEDs of the reaction product also shows the presence of nickel, it appears that nickel is forming a solid solution with iron and substituting at the iron lattice sites. Again there is no evidence of the reaction between Hi-Nicalon SiC fiber and the BN coating. The reaction observed between the uncoated, and nonuniform PBN and PBN/Si-rich PBN coated Hi-Nicalon SiC fibers and the RBSN matrix can explain the loss of the room-temperature fracture load. What is not still understood is the loss of the room-temperature load at the 1400 °C processing temperature for the BN/SiC coated Hi-Nicalon SiC fibers where no obvious microstructural changes at the coating/fiber interface occurred. We suspect that the reaction between impurities present in silicon powder and uncoated regions of the Hi-Nicalon SiC fibers is also the probable cause for strength degradation of these fibers.

SUMMARY OF RESULTS

Microstructural stability and room-temperature fracture properties of uncoated and three types of CVD coated Hi-Nicalon SiC fiber tows infiltrated with silicon and then nitrided at 1200 and 1400 °C for 4 hr in N_2 were determined. Major findings are as follows.

(1) The uncoated Hi-Nicalon fibers degrade under RBSN processing conditions. Significant reaction between the RBSN matrix and SiC fiber was observed. The iron impurity present in silicon powder and the nitride enhancing additive (NiO) used reacted with the fiber to form complex silicides.

(2) Both PBN and PBN/Si-rich PBN coated fibers showed a nonuniform coating thickness. The thickness of CVD coatings on the outer fibers in the tow were significantly greater than that of the fibers in the interior of the tow. In

the uncoated regions of the fibers, iron impurities and the NiO additive reacted with the SiC fibers to form silicides. Both coatings provided an improvement in room-temperature fracture load at the 1200 °C processing temperature. More uniform coating would provide better protection.

(3) The BN layer of the BN/SiC coated Hi-Nicalon SiC fiber tows is uniform. The BN layers appear to be stable at both processing temperatures. The outer SiC layer on top of the BN layer reacted with NiO, in the tow composites processed at 1400 °C. The BN/SiC coated Hi-Nicalon SiC fiber tows processed at 1200 °C yielded the highest room temperature fracture load, whereas those processed at 1400 °C showed significant loss in room-temperature load. The mechanism of degradation of fiber tows at higher processing temperature is not understood.

(4) All three types of CVD coatings are suitable for fabrication of Hi-Nicalon/RBSN composites.

CONCLUSION

Strong and tough SiC fiber-reinforced RBSN composites can be fabricated by using PBN, PBN/Si-rich PBN, or BN/SiC coated Hi-Nicalon SiC fibers provided that the coating is uniform and continuous. However to minimize fiber degradation caused by the reaction between exposed Hi-Nicalon SiC fibers and the nitride enhancing additive/iron impurities in commercial silicon powder, processing of the composite should be done at temperature less than 1400 °C.

REFERENCES

1. R.T. Bhatt, "Effects of Fabrication Conditions on the Properties of SiC Fiber Reinforced Reaction-Bonded Silicon Nitride Matrix Composites (SiC/RBSN)," NASA TM—88814 (1986).
2. J.W. Lucek, G.A. Rossetti, Jr., and S.D. Hartline, "Stability of Continuous Si-C (-O) Reinforcing Elements in Reaction-Bonded Silicon Nitride Process environments," pp. 27–38 in Metal Matrix, Carbon, and Ceramic matrix Composites 1985, NASA CP–2406, Edited by J.D. Buckley, NASA, Washington, D.C., 1985.
3. T.L. Starr, D.L. Mohr, W.J. Lackey, and J.A. Hanigofsky, "Continuous Fiber-Reinforced Reaction Sintered Silicon Nitride Composites," Cer. Eng. Sci. Proc., **14** [9–10] pp. 1125–1132 (1993).
4. G.H. Woblewska and G. Ziegler, "Reaction-Bonded Si_3N_4 Reinforced with Continuous SiC Fibers: Processing and Interface Characteristics," in High-Temperature Ceramic-Matrix Composites II: Manufacturing and Materials Development, Edited by A.G. Evans and R. Naslain, Ceramic Trans. **58**, pp. 131–136 (1995).
5. R.T. Bhatt and A. Garg, "Strength and Microstructural Stability of Hi-Nicalon SiC Fibers in N_2 and in Nitridation Environment," Proc. ICCM 10, Eds. A. Poursartip, and K. Street, **VI**, pp. 339–347 (1995).
6. T.P. Herbell, T.K. Glasgow and N.W. Orth, "Demonstration of a Silicon Nitride Attrition Mill for Production of Fine Pure Si and Si_3N_4 Powders," Bull. Amer. Ceram. Soc. **63** [9] (1984) pp. 1176.
7. R. Koch and A.F. Marshall, "Specimen Preparation for Transmission Electron Microscopy of Materials II," Mater. Res. Soc. Symp. Proc., 199, pp. 145–152 (1990).

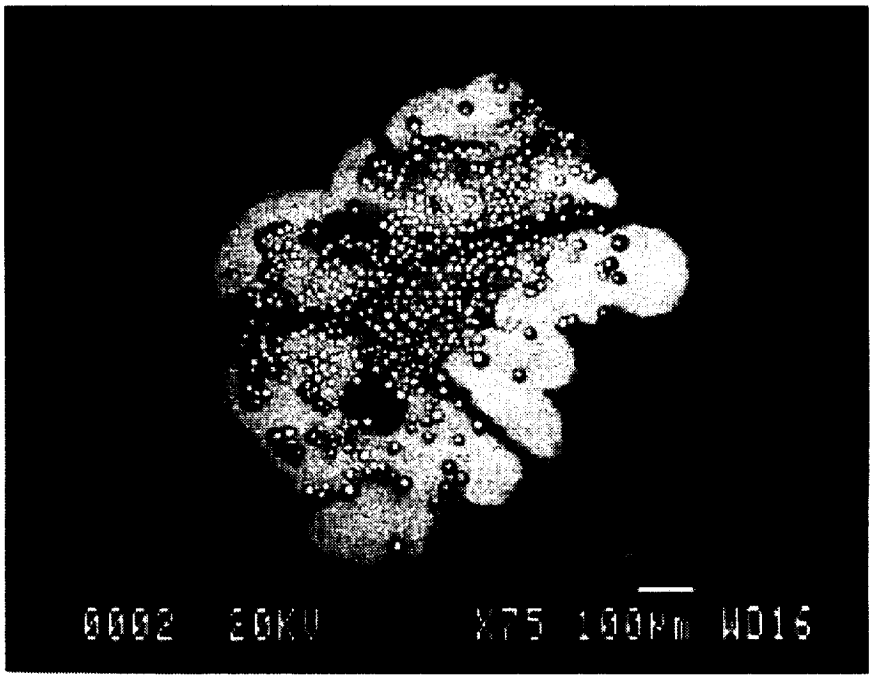


Figure 1.—SEM micrograph of the cross-section of the PBN/Si-rich PBN coated Hi-Nicalon/RBSN tow composite showing distribution of fibers.

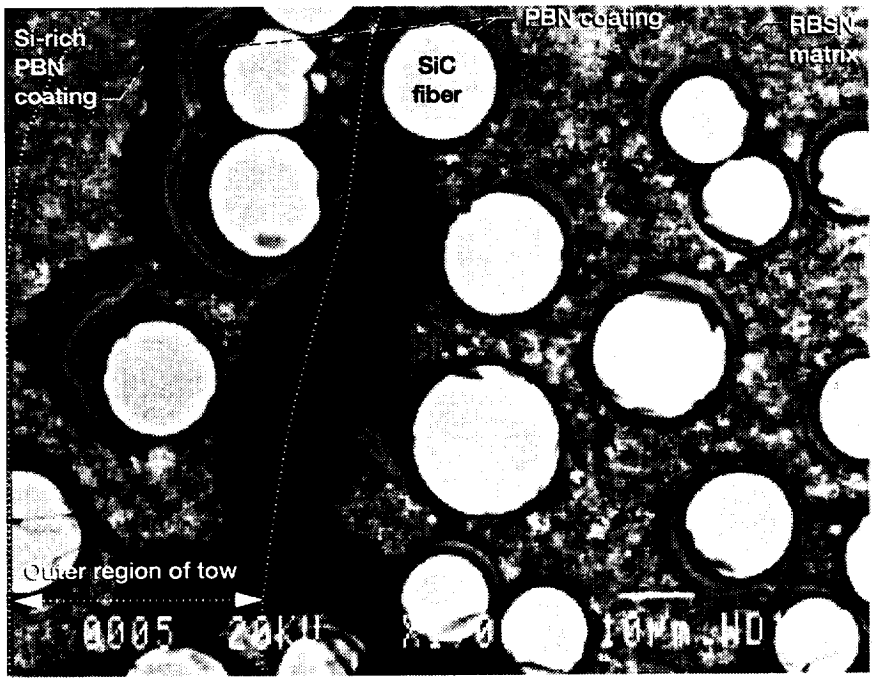


Figure 2.—SEM micrograph of the cross-section of the PBN/Si-rich PBN coated Hi-Nicalon/RBSN tow composite showing coating thickness variation within the tow.

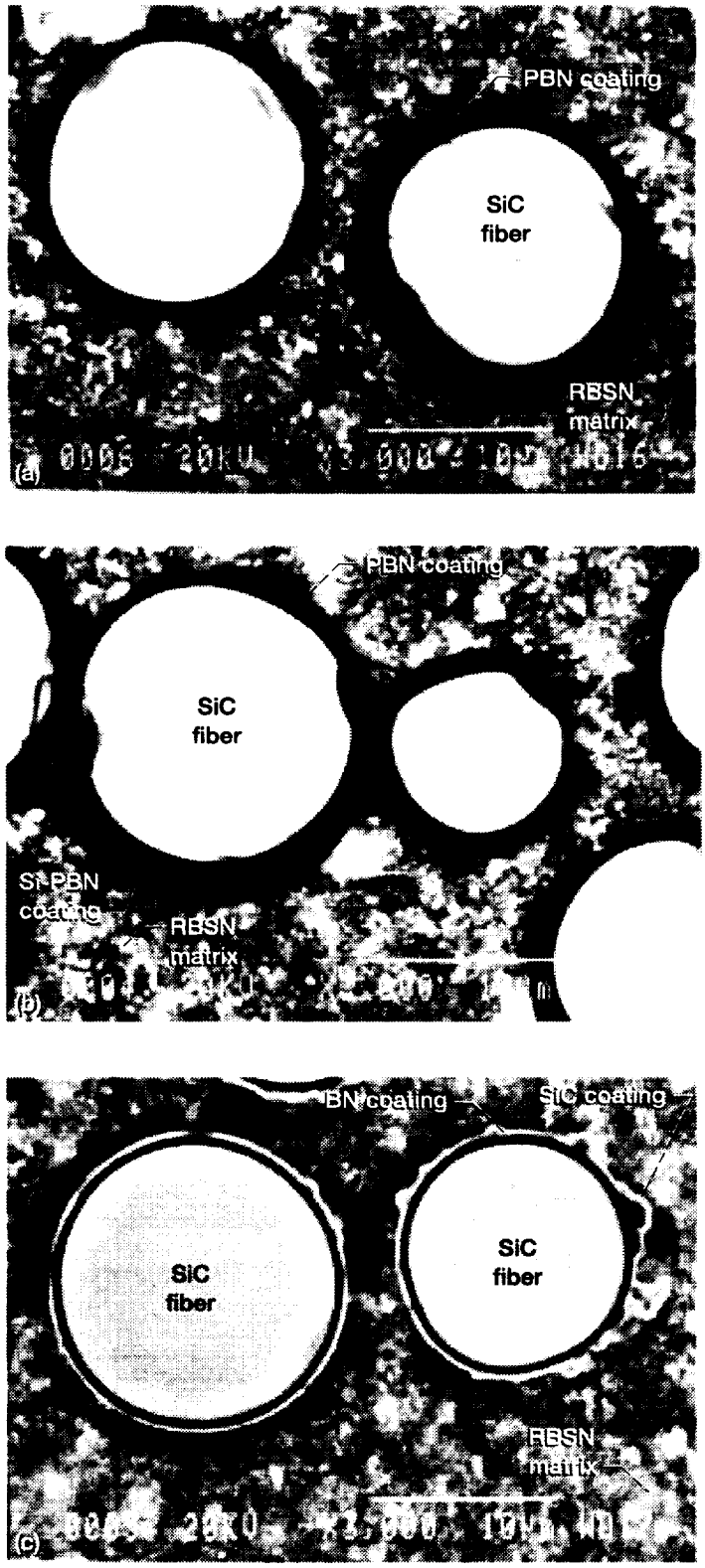


Figure 3.—SEM micrographs of the cross-section of the Hi-Nicalon/RBSN tow composites processed at 1200 °C in N_2 for 4 hr showing morphology of interface coating. (a) PBN coated. (b) PBN/Si-rich-PBN coated. (c) BN/SiC coated.

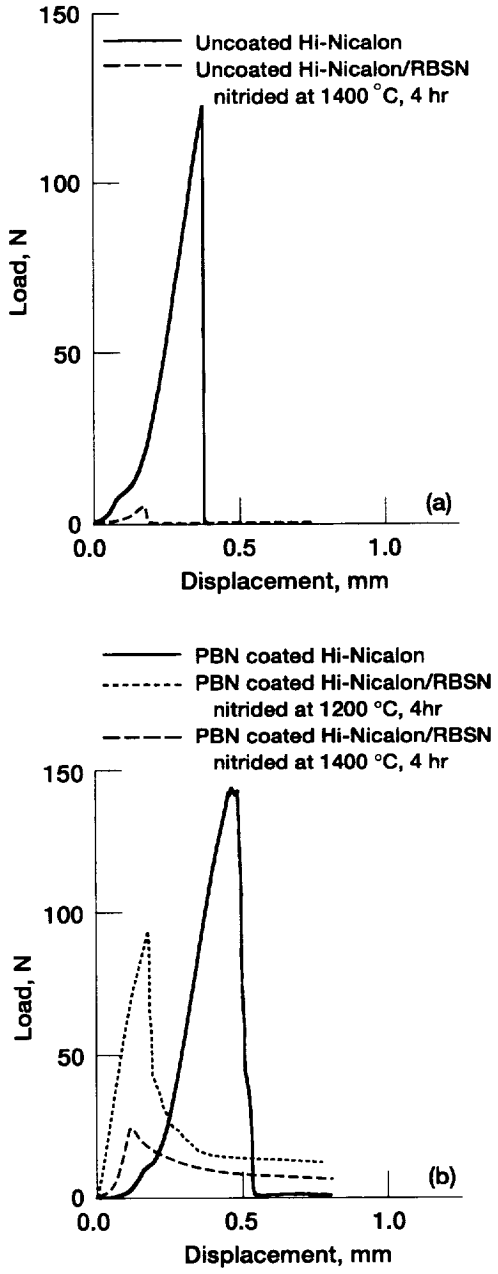


Figure 4.—Room temperature load-displacement curves for Hi-Nicalon SiC/RBSN tow composites. (a) Uncoated. (b) PBN coated.

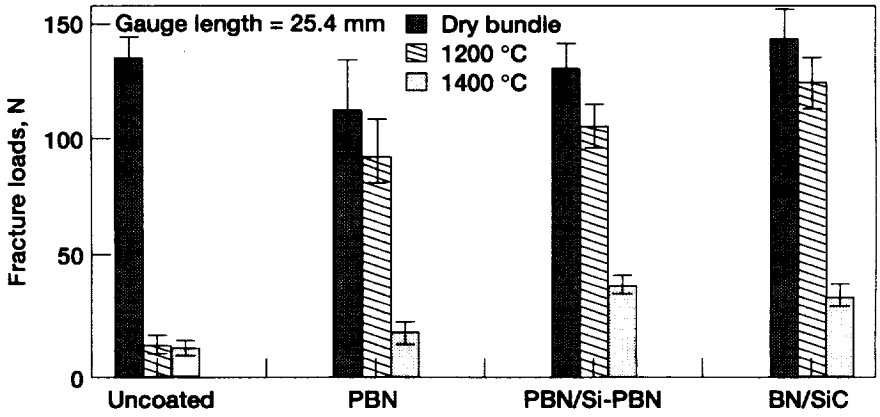
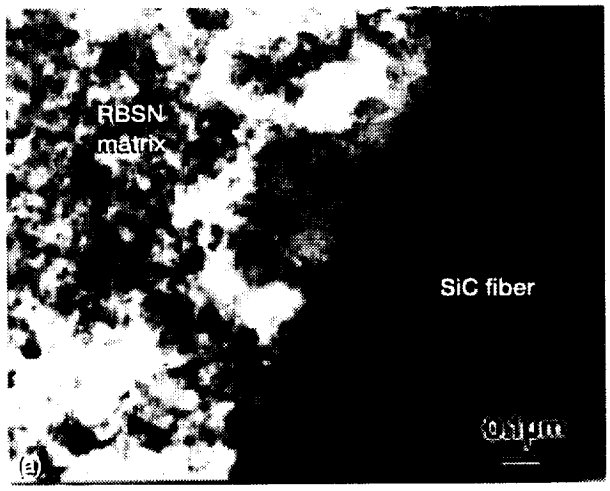
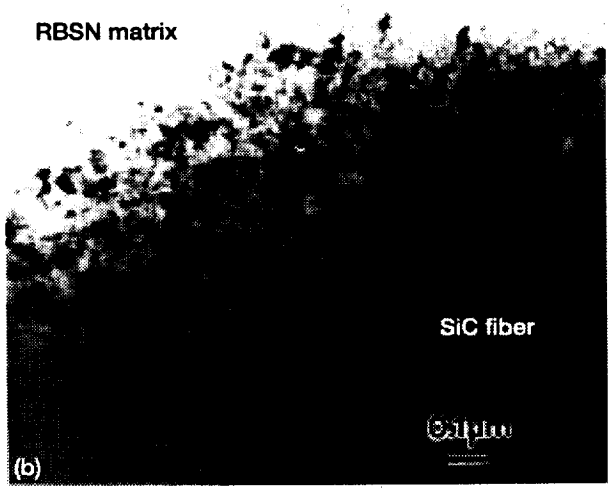


Figure 5.—Room temperature ultimate tensile fracture load of uncoated and CVD coated Hi-Nicalon SiC fiber tows and Hi-Nicalon/RBSN tow composites nitrided in N_2 for 4 hr.





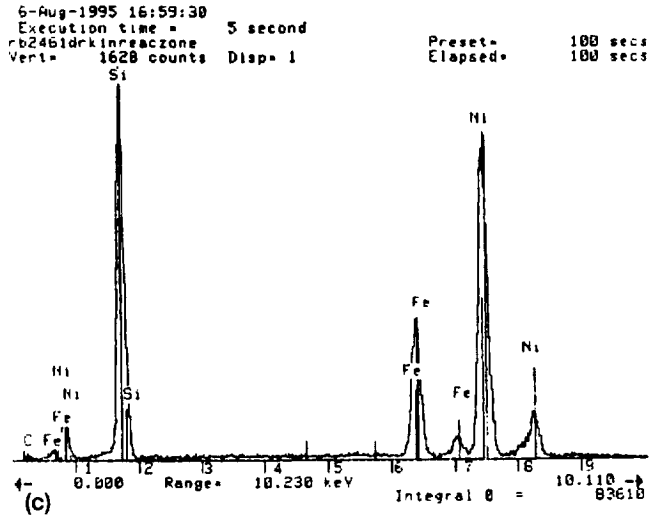


Figure 6.—TEM micrographs of the uncoated Hi-Nicalon/RBSN tow composites showing reaction between the fiber and RBSN matrix. (a) Nitrided in N_2 at 1200 °C for 4 hr. (b) Nitrided in N_2 at 1400 °C for 4 hr. (c) XEDS of the reaction zone in (b). Star indicates the region of the interface zone analyzed.

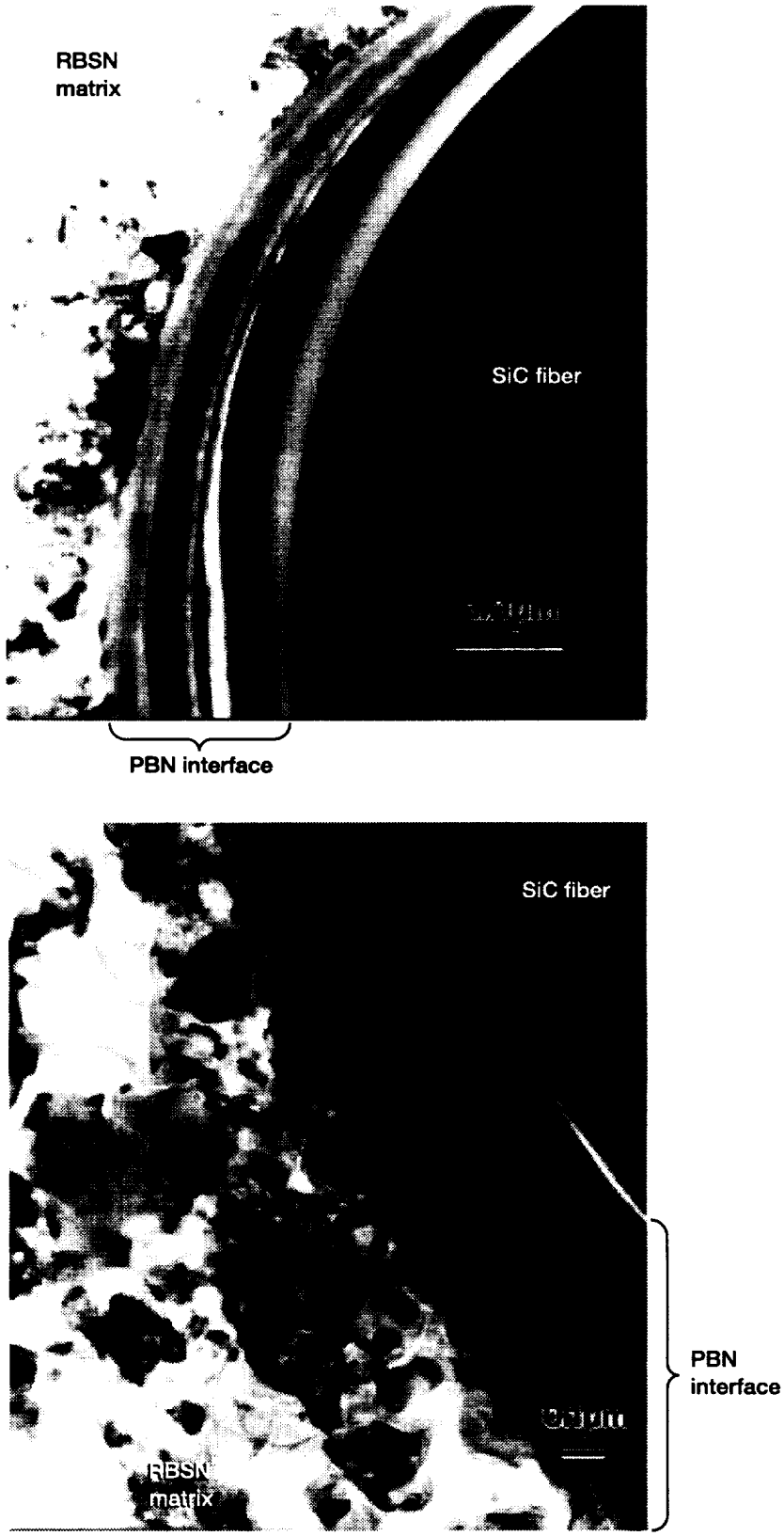


Figure 7.—TEM micrographs of the PBN coated Hi-Nicalon/RBSN tow composite nitrided at 1400 °C in N_2 for 4 hr showing no reaction between PBN coating and RBSN matrix.

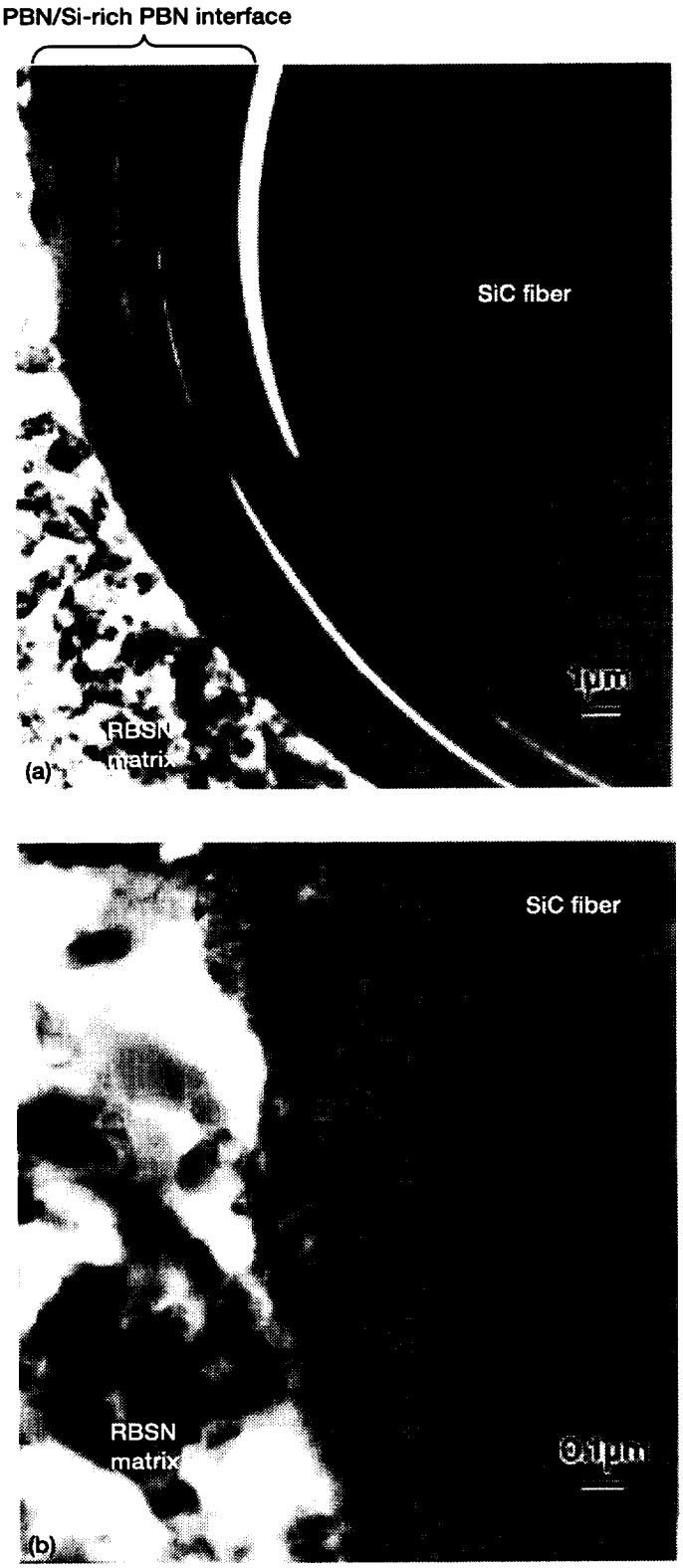


Figure 8.—TEM micrographs of the PBN/Si-PBN coated Hi-Nicalon/RBSN tow composite nitrided at 1400 °C in N_2 for 4 hr showing reaction between the fiber and RBSN matrix. (a) PBN/Si-rich PBN and RBSN interface. (b) Region of the uncoated fiber showing reaction with iron and nickel.

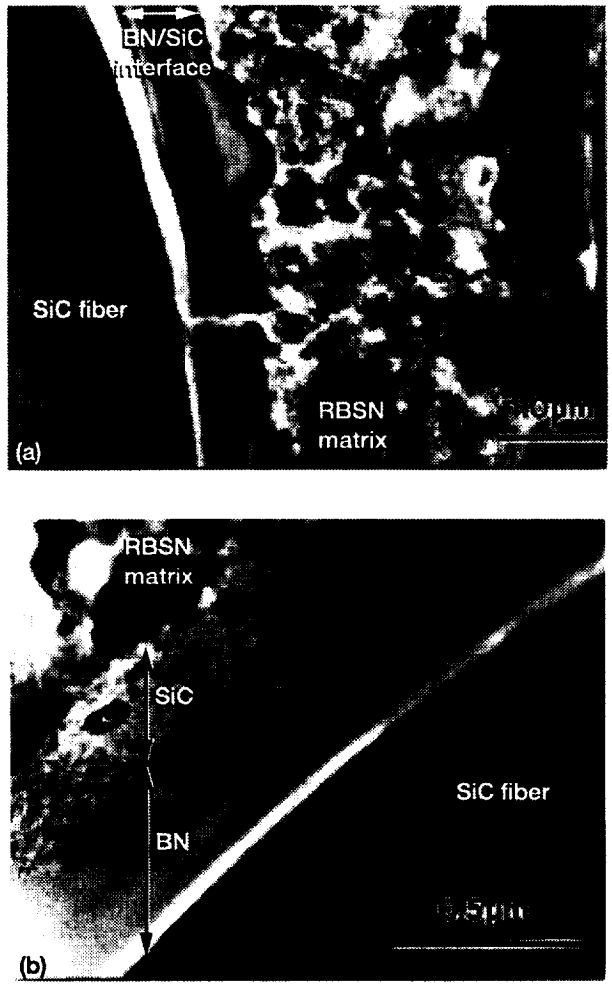


Figure 9.—TEM micrographs of the BN/SiC coated Hi-Nicalon/RBSN tow composite showing reaction between the fiber and RBSN matrix. (a) Nitrided in N_2 at 1200 °C for 4 hr. (b) Nitrided in N_2 at 1400 °C for 4 hr. (c) XEDS of the reaction zone in (b). Star indicates the region of the interface zone analyzed.

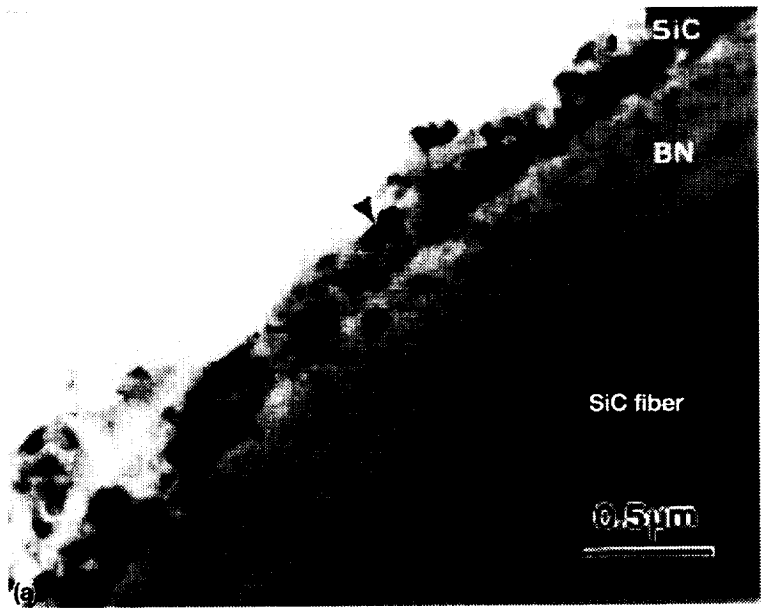
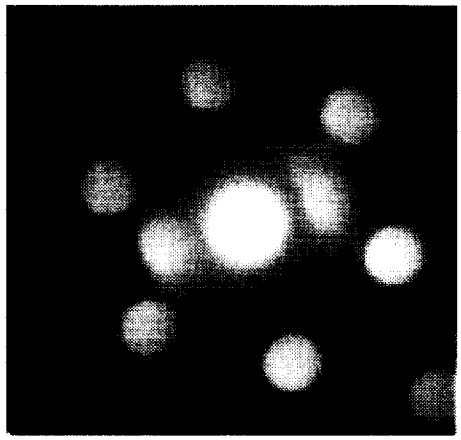
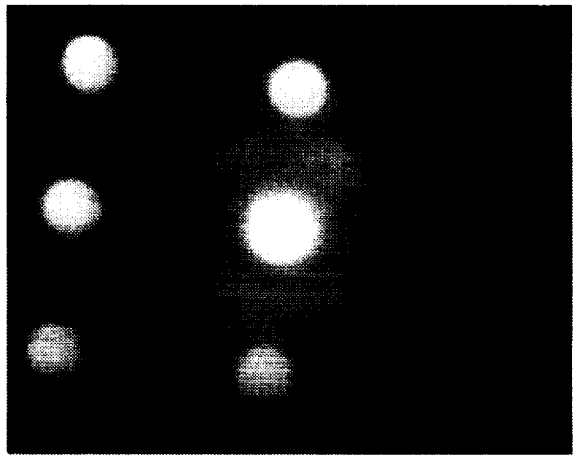


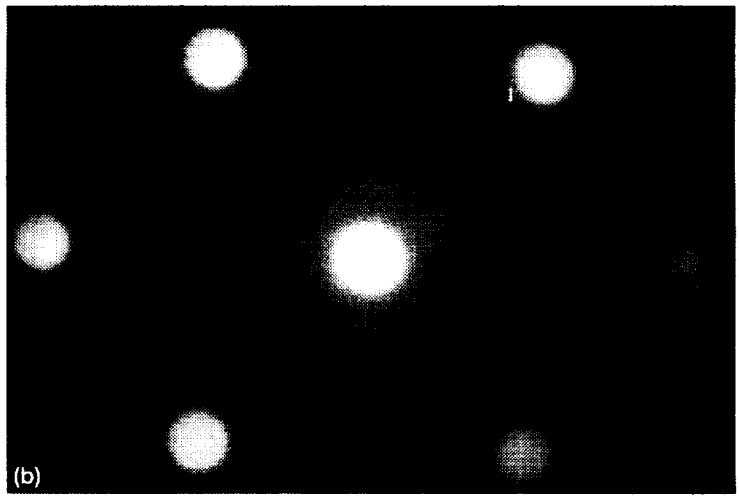
Figure 10.—TEM micrograph and CBED analysis of reaction product in BN/SiC coated Hi-Nicalon/RBSN tow composite nitrided in N_2 at 1400 °C for 4 hr. (a) TEM micrograph of the BN/SiC interface. (b) CEBD patterns of the particles within the SiC coating.



[111] Zone axis



[011] Zone axis



[153] Zone axis

Figure 10.—Concluded. (b) CEBD patterns of the particles within the SiC coating.

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13. ABSTRACT (*Maximum 200 words*)

Uncoated Hi-Nicalon silicon carbide (SiC) fiber tows and those coated with a single surface layer of pyrolytic boron nitride (PBN), double layers of PBN/Si-rich PBN, and boron nitride (BN)/SiC coatings deposited by chemical vapor deposition (CVD) method were infiltrated with silicon slurry and then exposed to N_2 to 4 hr at 1200 and 1400°C. Room temperature ultimate tensile fracture loads and microstructural characterization of uncoated and CVD coated Hi-Nicalon SiC fiber reinforced reaction-bonded silicon nitride (RBSN) tow composites were measured to select suitable interface coating(s) stable under RBSN processing conditions. Results indicate that room temperature ultimate fracture loads of the uncoated Hi-Nicalon SiC/RBSN tow composites nitrided at both temperatures were significantly lower than those of the uncoated Hi-Nicalon tows without slurry infiltration. In contrast, all CVD coated Hi-Nicalon SiC/RBSN tow composites retained a greater fraction of the dry tow fracture load after nitridation at 1200°C, but degraded significantly after nitridation at 1400°C. Reaction between metal impurities (Fe and Ni) present in the attrition milled silicon powder and uncoated regions of SiC fibers appears to be the probable cause for fiber degradation.

14. SUBJECT TERMS

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